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# *Indian Standard*

# SPECIFICATION FOR SODIUM THIOSULPHATE, CRYSTALLINE, PHOTOGRAPHIC GRADE

## ( *Fourth Revision* )

UDC 771·7 : 546·332·232

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 4

*September* 1986

# *Indian Standard*

# SPECIFICATION FOR SODIUM THIOSULPHATE, CRYSTALLINE, PHOTOGRAPHIC GRADE

## *( Fourth Revision )*

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# *Indian Standard*

# SPECIFICATION FOR SODIUM THIOSULPHATE, CRYSTALLINE, PHOTOGRAPHIC GRADE

## ( *Fourth Revision* )

## 0. FOREWORD

**0.1** This Indian Standard (Fourth Revision) was adopted by the Indian Standards Institution on 28 February 1986, after the draft finalized by the Photographic Materials Sectional Committee had been approved by the Chemical Division Council.

**0.2** This standard was first published in 1950 and subsequently revised in 1957, 1964 and 1972. This standard had earlier contained four grades, namely, technical, photographic, pure and analytical reagent, of the material. The Photographic Materials Committee responsible for the preparation of this standard felt that other grades of the material were not getting due attention in this Committee and, therefore, decided to revise the standard after retaining only photographic grade and transferring the other grades to Inorganic Chemicals (Misc) Sectional Committee. In the present revision, the limits for heavy metals and iron content have also been modified.

**0.3** Sodium thiosulphate, crystalline is commonly known as hypo and is used in the photographic industry as a fixing agent to dissolve unchanged silver salts from exposed negatives.

**0.4** For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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*Rules for rounding off numerical values ( *revised* ).

## 1. SCOPE

**1.1** This standard prescribes the requirements and the methods of sampling and test for sodium thiosulphate, crystalline, photographic grade.

## 2. REQUIREMENTS

**2.1 Description** — The material shall be in the form of colourless crystals or crystalline powder, free from extraneous impurities.

**2.2 Solution in Water** — A freshly prepared aqueous solution of the material containing 40 g of the material in 100 ml of carbon dioxide-free water shall be clear and free from sediment other than a slight flocculence.

**2.3** The material shall also comply with the requirements given in Table 1, when tested according to methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 4 of the table.

### TABLE 1 REQUIREMENTS FOR SODIUM THIOSULPHATE, CRYSTALLINE

| Sl No. | Characteristic | Requirement | Method of Test (Ref to Cl No. in Appendix A) |
|---|---|---|---|
| (1) | (2) | (3) | (4) |
| i) | Sodium thiosulphate ( $Na_2S_2O_3.5H_2O$ ), percent by mass | 99 to 101 | A-2 |
| ii) | *p*H of 10 percent solution | 6·5 to 8·5 | A-3 |
| iii) | Insoluble matter and metals other than alkali metals, percent by mass, *Max* | 0·2 | A-4 |
| iv) | Sodium sulphide ( as $Na_2S$ ), percent by mass, *Max* | 0·001 | A-5 |
| v) | Heavy metals ( as Pb ), percent by mass, *Max* | 0·001 | A-6 |
| vi) | Iron ( as Fe ), percent by mass, *Max* | 0·002 | A-7 |

## 3. PACKING AND MARKING

**3.1** The material shall be packed in suitable waterproof lined containers.

**3.2** The containers shall be securely closed after filling and marked with the manufacturer's name; mass of material in the container; recognized trade-mark, if any; and batch number or the month and year of manufacture.

**3.2.1** The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

## 4. SAMPLING

**4.1** Method of preparing the representative test samples and the criteria for judging the conformity to this standard shall be as prescribed in Appendix B.

# APPENDIX A

( *Clause* 2.3 )

## ANALYSIS OF SODIUM THIOSULPHATE, CRYSTALLINE, PHOTOGRAPHIC GRADE

## A-1. QUALITY OF REAGENTS

**A-1.1** Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

## A-2. DETERMINATION OF SODIUM THIOSULPHATE

**A-2.0 Principle of the Method** — The sodium thiosulphate content is determined by titrating a solution of the material with standard iodine solution, using starch as indicator.

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*Specification for water for general laboratory use (*second revision*).

### A-2.1 Reagents

**A-2.1.1** *Starch Solution* — Triturate 5 g of starch and 0·01 g of the mercuric iodide with 30 ml of water in a mortar. Pour the resulting paste into one litre of boiling water, boil for 3 minutes, allow the solution to cool and decant off the clear liquid.

**A-2.1.2** *Standard Iodine Solution* — 0·1 N (*see* IS : 2316-1968*).

**A-2.2 Procedure** — Weigh accurately about one gram of the material and dissolve in 50 ml of water. Titrate the solution with standard iodine solution using starch solution as indicator, the end point being indicated by the appearance of blue colour.

### A-2.3 Calculation

$$\text{Sodium thiosulphate } (Na_2S_2O_3.5H_2O), \text{ percent by mass} = \frac{24{\cdot}82\, VN}{M}$$

where

$V$ = volume in ml of standard iodine solution used in the titration,

$N$ = normality of standard iodine solution, and

$M$ = mass in g of the material taken for the test.

## A-3. DETERMINATION OF *p*H OF 10 PERCENT SOLUTION

**A-3.0 General** — *p*H shall be determined by using a *p*H meter as prescribed in **A-3.1** or by the indicator method given in **A-3.2**; but in case of dispute, the *p*H meter method shall be adopted.

### A-3.1 *p*H Meter Method

**A-3.1.1** *Procedure* — Dissolve 5 g of the material in freshly boiled and cooled water, dilute to 50 ml and mix. Determine the *p*H value of the solution using a *p*H meter with glass electrode.

### A-3.2 Indicator Method

**A-3.2.1** *Indicators* — A list of indicators suitable for the determination of *p*H value is given below:

| | |
|---|---|
| a) Thymol blue (acid range) | *p*H 1·2 to 2·8, red to yellow |
| b) Bromophenol blue | *p*H 2·8 to 4·6, yellow to blue violet |

---

*Methods of preparation of standard solutions for colorimetric and volumetric analysis (*first revision*).

| | |
|---|---|
| c) Bromocresol green | *p*H 3·6 to 5·2, yellow to blue |
| d) Methyl red | *p*H 4·2 to 6·3, red to yellow |
| e) Bromocresol purple | *p*H 5·2 to 6·8, yellow to blue violet |
| f) Bromothymol blue | *p*H 6·0 to 7·6, yellow to blue |
| g) Phenol red | *p*H 6·8 to 8·4, yellow to red |
| h) Cresol red | *p*H 7·2 to 8·8, yellow to red |
| j) Thymol blue (alkali range) | *p*H 8·0 to 9·6, yellow to blue |

**A-3.2.2** Standard calibrated glass discs may also be used for comparison.

**A-3.2.3** *Test Temperature* — The test shall be carried out at 27 ± 2°C.

**A-3.2.4** *Procedure* — In a hard-glass tube, place 10 ml of the solution prepared by dissolving 10·0 g of the material in 100 ml of carbon dioxide-free water. Determine the approximate *p*H by using a universal indicator. Repeat using a solution of the indicator ( about one twentieth of the volume of the liquid being tested ) which corresponds to the approximate *p*H found. Compare the colour produced with a series of buffer tubes of known *p*H, each containing the same proportion of the indicator.

**A-3.2.5** The *p*H of the buffer solution, which matches with that of the material, shall be reported as *p*H of 10 percent solution of the material.

## A-4. DETERMINATION OF INSOLUBLE MATTER AND METALS OTHER THAN ALKALI METALS

**A-4.0 Principle of the Method** — Insoluble matter and metals other than alkali metals are determined by mixing the material with water, adding to the solution ammonium oxalate, ammonium phosphate and ammonium hydroxide and weighing the residue obtained on the filter.

### A-4.1 Reagents

**A-4.1.1** *Ammonium Oxalate Solution* — 4 percent.

**A-4.1.2** *Ammonium Phosphate Solution* — 10 percent.

**A-4.1.3** *Dilute Ammonium Hydroxide Solution* — 1 : 9 (*v*/*v*).

**A-4.1.4** *Dilute Ammonium Hydroxide Solution* — 1 : 39 (*v*/*v*).

**A-4.2 Procedure** — Dissolve about 10 g of the material, accurately weighed, in 75 ml of water. Add 5 ml of ammonium oxalate solution, 2 ml of ammonium phosphate solution and 10 ml of dilute ammonium hydroxide (1 : 9). Allow it to stand overnight. Filter off the precipitate formed through an ashless filter paper and wash with dilute ammonium hydroxide (1 : 39). Dry the residue, ignite at about 600°C, cool in a desiccator and weigh.

### A-4.3 Calculation

$$\text{Insoluble matter and metals other than alkali metals, percent by mass} = 100\,\frac{M_1}{M_2}$$

where

$M_1$ = mass in g of the ignited residue, and

$M_2$ = mass in g of the material taken for the test.

## A-5. TEST FOR SODIUM SULPHIDE

**A-5.0 Principle of the Method** — Colour produced with alkaline lead acetate solution is compared with the colour produced with standard sulphide solution under identical conditions.

### A-5.1 Apparatus

**A-5.1.1** *Nessler Cylinders* — 50-ml capacity.

### A-5.2 Reagents

**A-5.2.1** *Sodium Hydroxide Solution* — 10 percent.

**A-5.2.2** *Lead Acetate Solution* — 10 percent.

**A-5.2.3** *Alkaline Lead Acetate Solution* — Add sufficient amount of sodium hydroxide solution to lead acetate solution to dissolve the precipitate and provide a slight excess of sodium hydroxide.

**A-5.2.4** *Standard Sodium Sulphide* — Dissolve about 15 g of sodium sulphide ($Na_2S.9H_2O$) in water and dilute to 500 ml in a volumetric flask. Pipette out 20 ml of the solution in a conical flask and add 50 ml of 0·1 N iodine solution to it. Keep for 5 minutes and titrate with 0·1 N sodium thiosulphate solution. Apply a correction for the blank determination. One millilitre of 0·1 N iodine solution corresponds to 0·012 0 g of sodium sulphide ($Na_2S.9H_2O$). Dilute in a volumetric flask a volume of sodium sulphide solution containing 3·075 g of sodium sulphide ($Na_2S.9H_2O$) to 1 000 ml. Further dilute 10 ml of this solution to 1 000 ml in a volumetric flask. One millilitre of the diluted solution contains 0·01 mg of sodium sulphide (as $Na_2S$).

**A-5.3 Procedure** — Dissolve 2·00 g of the material in 10 ml of water and add 0·5 ml of alkaline lead acetate solution. Transfer to a Nessler cylinder and dilute to 50 ml and mix well. Carry out a control test in another Nessler cylinder using 2 ml of standard sulphide solution in place of the material.

**A-5.3.1** The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced with the material is not greater than that in the control test.

## A-6. TEST FOR HEAVY METALS

**A-6.0 Principle of the Method** — After oxidizing the thiosulphate with hydrogen peroxide, the colour produced by heavy metals with hydrogen sulphide is compared with that obtained with a standard solution of heavy metals.

### A-6.1 Apparatus

**A-6.1.1** *Nessler Cylinders* — 50-ml capacity.

### A-6.2 Reagents

**A-6.2.1** *Dilute Ammonium Hydroxide* — approximately 5 N.

**A-6.2.2** *Hydrogen Peroxide* — approximately 15 percent by mass.

**A-6.2.3** *Dilute Hydrochloric Acid* — approximately 5 N and 1 : 99 (*v/v*).

**A-6.2.4** *p-Nitrophenol Indicator* — Dissolve 0·2 g of *p*-nitrophenol in hot water and dilute with water to 100 ml.

**A-6.2.5** *Standard Lead Solution* — Dissolve 1·60 g of lead nitrate [$Pb(NO_3)_2$] in water, add one millilitre of concentrated nitric acid and make up the volume to 1 000 ml. When required for use, pipette out 10 ml of this solution and dilute to 1 000 ml with water. One millilitre of this solution contains 0·01 mg of lead (as Pb).

**A-6.2.6** *Hydrogen Sulphide Water* — freshly prepared, saturated, aqueous solution of hydrogen sulphide.

**A-6.3 Procedure** — Dissolve 5·0 g of the material in 20 ml of water. Add 5 ml of dilute ammonium hydroxide and then add slowly, in several portions, 40 ml of hydrogen peroxide. Allow to stand for 10 minutes. Evaporate to dryness on a steam-bath. Add 5 ml of dilute hydrochloric acid ( 5N ) and 10 ml of water. Heat to boiling. Dilute to 100 ml with water. Transfer 20 ml of the solution in a Nessler cylinder and neutralize with dilute ammonium hydroxide using *p*-nitrophenol as indicator. Add dilute hydrochloric acid (1 : 99) dropwise until neutral to the indicator and then 1 ml of acid in excess. Carry out a control test, following the same procedure, in another Nessler cylinder using 3 ml of standard lead solution in place of the material.

Add 10 ml of hydrogen sulphide water to each Nessler cylinder, dilute to the mark and shake the contents well. Compare the colour produced in the two cylinders.

**A-6.3.1** The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced in test with the material is not greater than that produced in the control test.

## A-7. TEST FOR IRON

**A-7.0 Principle of the Method** — Ferric iron gives a red colour with potassium thiocyanate. The colour produced by the material after oxidation is compared with the colour formed by a standard iron solution.

### A-7.1 Apparatus

**A-7.1.1** *Nessler Cylinders* — 50 ml capacity.

### A-7.2 Reagents

**A-7.2.1** *Concentrated Hydrochloric Acid* — conforming to IS : 265-1976*.

**A-7.2.2** *Ammonium Persulphate or Potassium Persulphate*

**A-7.2.3** *Butanolic Potassium Thiocyanate Solution* — Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient *n*-butanol to make up to 100 ml and shake vigorously until the solution is clear.

**A-7.2.4** *Standard Iron Solution* — Dissolve 0·702 g of ferrous ammonium sulphate [$FeSO_4(NH_4)_2SO_4.6H_2O$] in 10 ml of dilute sulphuric acid (10 percent, *v/v*) and dilute with water to 1 000 ml. Pipette out 10 ml of the solution and dilute with water to make up the volume to 100 ml. One millilitre of this solution contains 0·01 mg of iron (as Fe).

**A-7.3 Procedure** — Pipette out 20 ml of the solution prepared in **A-6.3** to a Nessler cylinder. Add 1 ml of concentrated hydrochloric acid, 30 mg of ammonium persulphate or potassium persulphate and 15 ml of butanolic potassium thiocyanate solution. Carry out a control test using 5 ml of standard iron solution in place of the material. Shake both the solutions in the cylinders vigorously for 30 seconds and allow the liquid to separate.

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*Specification for hydrochloric acid ( *second revision* ).

**A-7.3.1** The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of any red colour produced in the butanolic layer of the first Nessler cylinder is not greater than that produced in the control test.

# APPENDIX B

( *Clause* 4.1 )

## SAMPLING AND CRITERIA FOR CONFORMITY

### B-1. GENERAL REQUIREMENTS OF SAMPLING

**B-1.0** In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

**B-1.1** Samples shall not be taken at a place exposed to weather.

**B-1.2** Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

**B-1.3** To draw a representative sample, the contents of each container selected for sampling shall be mixed thoroughly by suitable means.

**B-1.4** The samples shall be placed in suitable, clean, dry and air-tight opaque glass containers.

**B-1.5** The sample containers shall be of such a size that they are almost completely filled by the sample.

**B-1.6** Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

**B-1.7** Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

### B-2. SCALE OF SAMPLING

**B-2.1 Lot** — All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the group of containers in each batch shall constitute separate lots.

**B-2.2** From each lot five containers shall be selected ( *see* **B-2.2.1** ) for drawing samples for test. In case the lot contains less than five containers, each container shall be tested.

**B-2.2.1** Arrange the containers in a lot in a systematic way and starting from any container, count them as 1, 2, 3...up to $r$ and so on. Every $r$th container thus counted shall be separated for drawing samples for test, where

$$r = \frac{\text{Number of containers in the lot}}{5}$$

NOTE — In case the value of $r$ comes out to be a fractional number it shall be taken as equal to the integral part of that value.

## B-3. TEST SAMPLES

**B-3.1 Drawing of Samples** — Draw a representative sample from each container selected.

**B-3.1.1** Mix portions of the material drawn from the same container to prepare the test sample representing the container. The quantity of material to be drawn from each container shall be sufficient to conduct all the tests given in **B-4** and shall not be less than 200 g.

**B-3.1.2** The samples shall be stored in dry opaque glass bottles or other suitable containers, sealed air-tight and labelled with all the particulars of sampling given in **B-1.6**.

**B-3.1.3** Five test samples representing the five containers selected shall constitute a set of test samples.

## B-4. NUMBER OF TESTS

**B-4.1** Tests for the determination of sodium thiosulphate, heavy metals and iron shall be conducted on each of the five test samples ( *see* **B-3.1.3** ). Tests for all other characteristics prescribed in **2** shall be conducted on the composite sample ( *see* **B-4.1** ).

## B-5. CRITERIA FOR CONFORMITY

### B-5.1 For Tests Carried Out on Five Test Samples

**B-5.1.1** *Sodium Thiosulphate* — From the test results for sodium thiosulphate, the mean ( $\bar{X}$ ) and range ( $R$ ) of test results shall be calculated ( range being defined as the difference between the maximum and the minimum values of test results ). The lot shall be considered to have passed if the values of the expression ( $\bar{X} \pm 0{\cdot}86\ R$ ) are within the limits prescribed in Table 1.

**B-5.1.2** *Heavy Metals and Iron* — The lot shall be considered to have passed for the characteristics tested ( *see* **B-4.2** to **B-4.4** ) if each test sample passes the corresponding tests prescribed.

**B-5.2 Tests Carried Out on Composite Test Sample** — The lot shall be considered to have passed in respect of the characteristics tested on the composite test sample ( *see* **B-4.1** to **B-4.4** ) if the composite test sample satisfies the corresponding tests prescribed.

**B-5.3** The lot shall be considered as conforming to the specification if it satisfies all the criteria given in **B-5.1** and **B-5.2.**

# INTERNATIONAL SYSTEM OF UNITS ( SI UNITS )

## Base Units

| QUANTITY | UNIT | SYMBOL |
|---|---|---|
| Length | metre | m |
| Mass | kilogram | kg |
| Time | second | s |
| Electric current | ampere | A |
| Thermodynamic temperature | kelvin | K |
| Luminous intensity | candela | cd |
| Amount of substance | mole | mol |

## Supplementary Units

| QUANTITY | UNIT | SYMBOL |
|---|---|---|
| Plane angle | radian | rad |
| Solid angle | steradian | sr |

## Derived Units

| QUANTITY | UNIT | SYMBOL | DEFINITION |
|---|---|---|---|
| Force | newton | N | 1 N = 1 kg.m/$s^2$ |
| Energy | joule | J | 1 J = 1 N.m |
| Power | watt | W | 1 W = 1 J/s |
| Flux | weber | Wb | 1 Wb = 1 V.s |
| Flux density | tesla | T | 1 T = 1 Wb/$m^2$ |
| Frequency | hertz | Hz | 1 Hz = 1 c/s ($s^{-1}$) |
| Electric conductance | siemens | S | 1 S = 1 A/V |
| Electromotive force | volt | V | 1 V = 1 W/A |
| Pressure, stress | pascal | Pa | 1 Pa = 1 N/$m^2$ |

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| 'F' Block, Unity Bldg, Narasimharaja Square, BANGALORE 560002 | 22 48 05 |
| Gangotri Complex, 5th Floor, Bhadbhada Road, T. T. Nagar, BHOPAL 462003 | 6 67 16 |
| Plot No. 82/83, Lewis Road, BHUBANESHWAR 751002 | 5 36 27 |
| 53/5, Ward No. 29, R. G. Barua Road, 5th Byelane, GUWAHATI 781003 | — |
| 5-8-56C L. N. Gupta Marg (Nampally Station Road), HYDERABAD 500001 | 23 10 83 |
| R14 Yudhister Marg, C Scheme, JAIPUR 302005 | 6 34 71<br>6 98 32 |
| 117/418 B Sarvodaya Nagar, KANPUR 208005 | 21 68 76<br>21 82 92 |
| Patliputra Industrial Estate, PATNA 800013 | 6 23 05 |
| Hantex Bldg (2nd Floor), Rly Station Road, TRIVANDRUM 695001 | 7 66 37 |
| *Inspection Offices* (With Sale Point): | |
| Pushpanjali 205-A West High Court Road, Bharampeth Extension, NAGPUR 440010 | 2 51 71 |
| Institution of Engineers (India) Building, 1332 Shivaji Nagar, PUNE 411005 | 5 24 35 |

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